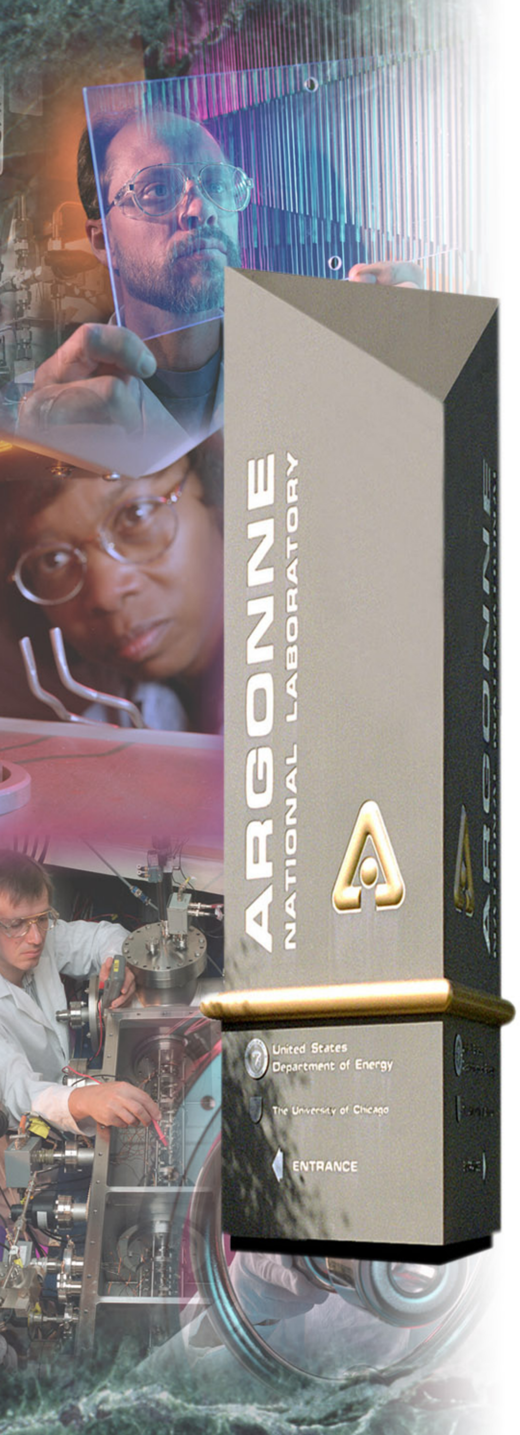


Report from the Workshop on Science with High-Energy X-rays

Dean R. Haeffner
XOR/APS

Presented at the APS Strategic Planning Meeting
Fontana, Wisconsin

September 2, 2004

Argonne National Laboratory

Office of Science
U.S. Department of Energy

A U.S. Department of Energy
Office of Science Laboratory
Operated by The University of Chicago

High-Energy X-rays: Why should anyone care?

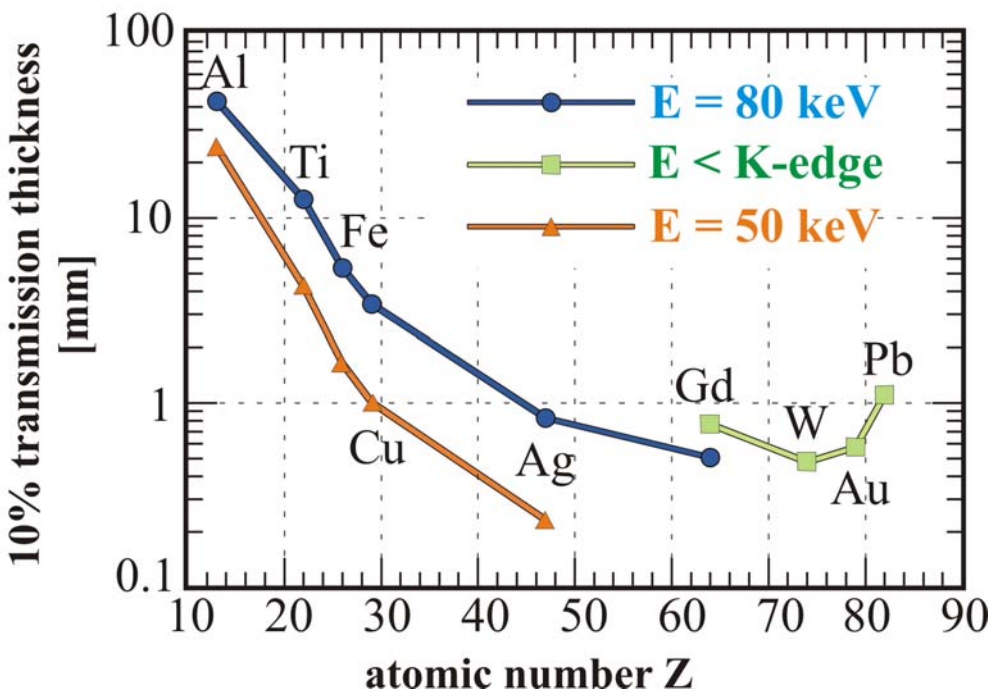
My definition ⬋ Photons between 35-200 keV

50 - 90 keV

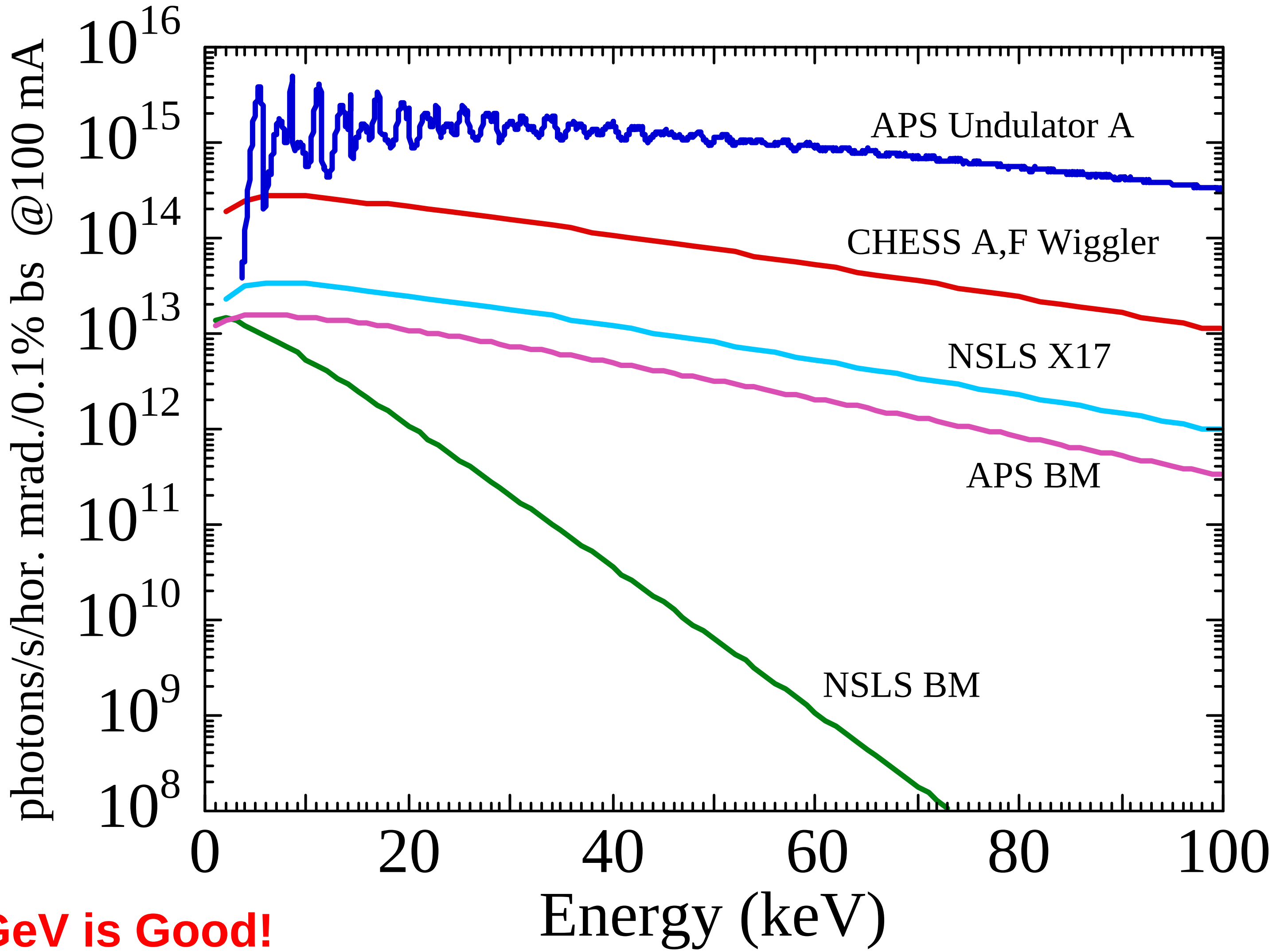
- **Low Absorption**
  - Bulk measurements
  - Special environments
  - Often comparable to neutrons

- **Simplified Scattering Processes**
  - Kinematical diffraction
  - Small absorption, polarization, & dispersion corrections

- **Small Diffraction Angles**
  - Large Q range



High-Energy X-rays: Why at the APS?

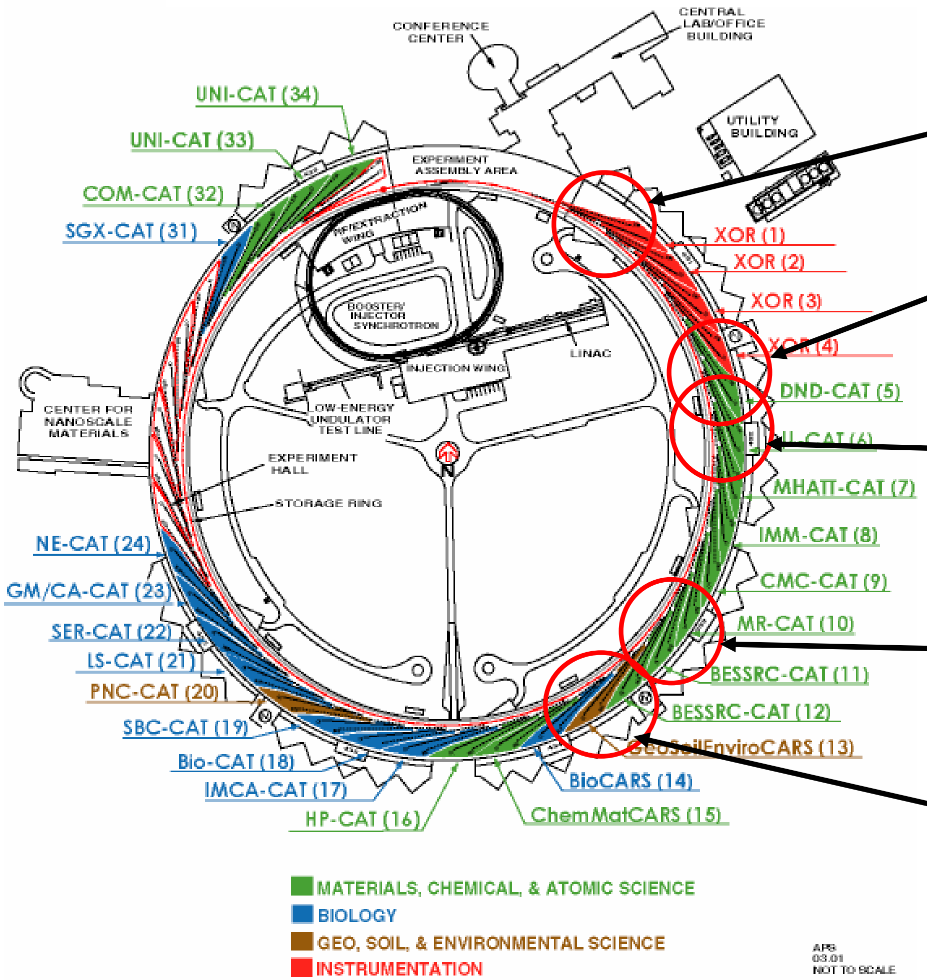


7 GeV is Good!

APS High Energy X-ray Capabilities

APS Collc

No optimized dedicated high-energy x-ray beamline



1-ID (XOR)
- Undulator
- High Brilliance Optics

5-BM (DND-CAT)
- Unoptimized bending magnet

6-ID (mu-CAT)
- High-energy side station

11-ID (BESSRC-CAT)
- Elliptical multipole wiggler
- Triple-axis diffractometer

13-ID, 13-BM (GSECARS-CAT)
- Geology/Earth Sciences
- Tomography

Workshop on Science with High-Energy X-rays

Held August 9, 10 at the APS

Local Organizing Committee:

Jon Almer (APS)

Mark Beno (APS)

Peter Lee (APS)

Ulrich Lienert (APS)

Doug Robinson (Ames Lab/MU-CAT)

Scientific Program Advisory Committee:

Ersan Üstündag (Iowa St.)

Bob Suter (Carnegie Mellon)

Dorte Juul Jensen *(RISØ)*

Angus Wilkinson (Georgia Tech)

Takeshi Egami (U. of Tenn.)

1. **Explore new and emerging scientific and technological areas defined in the scope of this workshop.**
2. **Broaden the community interaction by including researchers from various methodologies (e.g., EM, neutron scattering, etc.)**
3. **Identify new scientific proposals/programs specific to the emerging areas which can benefit from the use of High–Energy X-rays that the participants will bring to the APS during next 5 to 10 years. Also evaluate the capital and operational requirements for these proposals/programs.**
4. **In addition to available beamline capabilities at the APS, identify future needs to support research in this area of science and technology.**
5. **Address the need and support for theoretical work to strengthen the experimental research.**
6. **Prepare a summary document for the archival literature to serve as a roadmap for the future applications of high-energy x-rays and suggest the role of the Advanced Photon Source towards this objective.**

Workshop Program

Monday, August 9

AM:

- **Plenary Sessions (A1100)**
- **Lunch (5th Floor Gallery)**

PM:

- **Plenary Sessions (A1100)**
- **Group Photo**
- **Tours/Posters**
- **6:30 No Host Dinner (ANL Guest House)**

Tuesday, August 10

AM:

- **Parallel Sessions (A: A1100, B: A5000)**
- **Lunch (5th Floor Gallery)**

PM:

- **Parallel Sessions (A: A1100, B: A5000)**
- **Parallel Breakout Discussions**
  - **A: Ersan Üstündag (Discussion Leader)**
  - **B: Angus Wilkinson (Discussion Leader)**
- **Joint Summary Session (A1100)**

Workshop on Science with High-Energy X-rays

<u>Topics</u>

Optimized High Energy X-ray Beamline

Structural Science

- **Powder Diffraction**
- **PDF**
- **Diffuse Scattering**
- **Electrostatic Levitation**

Mechanical Behavior of Materials

- **Polycrystalline Stress/Strain**
- **Embedded grain studies (3D XRDM)**
- **Texture**

High Energy Small Angle Scattering

High Energy Absorption Spectroscopy

Atomic Physics

Industrial Applications

Actinide Science

Magnetic Scattering

Approximately ~45 attendees

Physicists

Materials Scientists

Environmental/Geo

Theory

National Labs

Industry

Universities

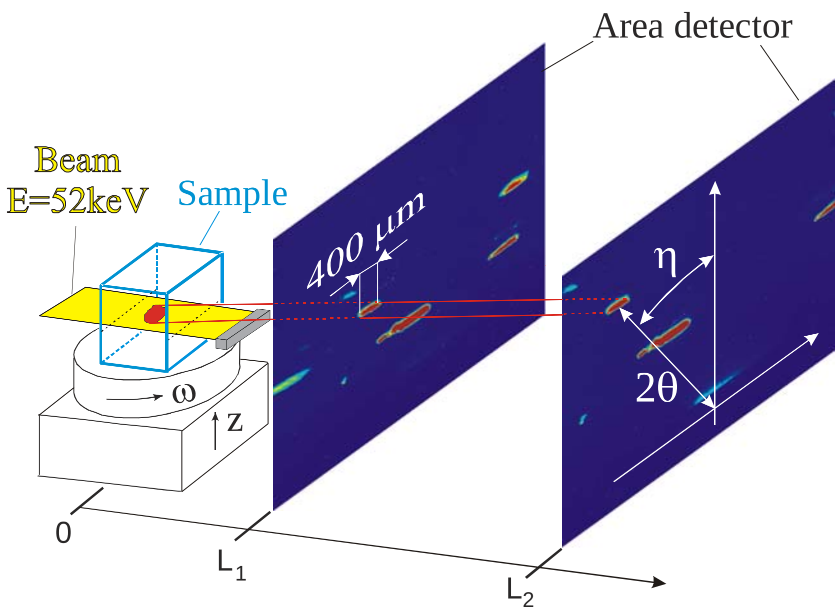
Speakers

Dean Haeffner (APS)
Sarvjit Shastri (APS)
John Parise (SUNY-Stoneybrook)
Ersan Üstündag (Iowa St./Ames Lab.)
Greg Rohrer (Carnegie Mellon)
Dorte Juul Jensen (Risø)
Andrew Allen (NIST)
Ronald Frahm (Wuppertal)
Elliot Kantor (ANL)
Yan Gao (GE)
Valeri Petkov (C. Michigan)
Mark Daymond (Queen's Univ.)
Anke Pyzalla (Tech. U. Wien)
Rosa Barabash (Oak Ridge)

Wolfgang Pantleon (Risø)
Todd Hufnagel (Johns Hopkins)
Mark Bourke (Los Alamos)
Rudy Wenk (Cal. Berkeley)
Paul Dawson (Cornell)
Angus Wilkinson (Georgia Tech.)
Brian Toby (NIST)
Jon Hanson (BNL)
Peter Chupas (ANL)
Matt Kramer (Iowa St./Ames Lab.)
Lynn Soderholm (ANL)
Alan Goldman (Iowa St./Ames Lab.)
Jorg Strempfer (Max Planck-Stuttgart)
Ray Osborn (ANL)

Science Examples: 3D X-ray Diffraction Microscope

- Grain position, grain boundary topology
- Crystallographic phase & orientation

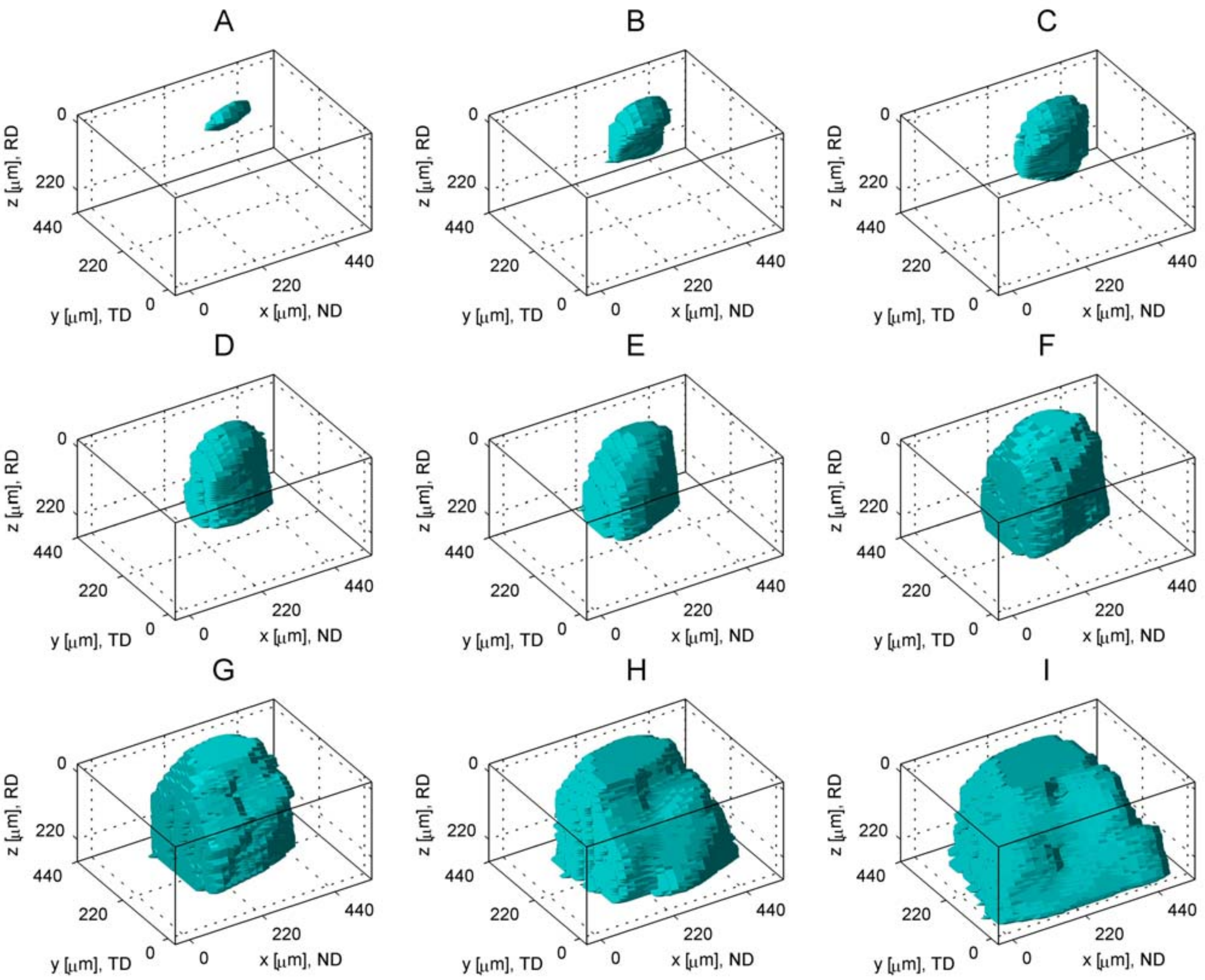


- Line focus
- Reflections by ω-rotation
- Projects grain cross section onto detector
- Backtracking => grain outline
- Grain orientation
- Some minutes per layer
- Limitation: mosaic spread

- Grain growth
- Phase transformation
- Initial state before processing

H.F. Poulsen *et al.*, J. Applied Cryst., 2001

Recrystallization



Growth of an Al grain inside the bulk

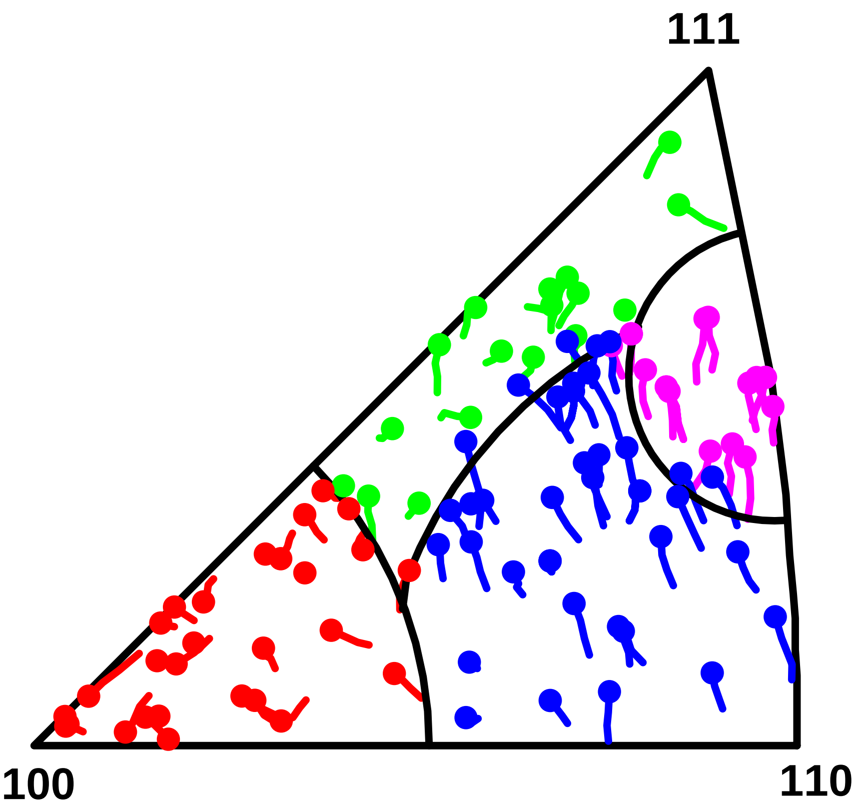
From Jensen

Schmidt, S., Nielsen, S.F., Gundlach, G., Margulies, L., Huang, X., Juul Jensen, D., Science, 2004, 229-232.

ESRF ID11

Grain Rotations under Loading

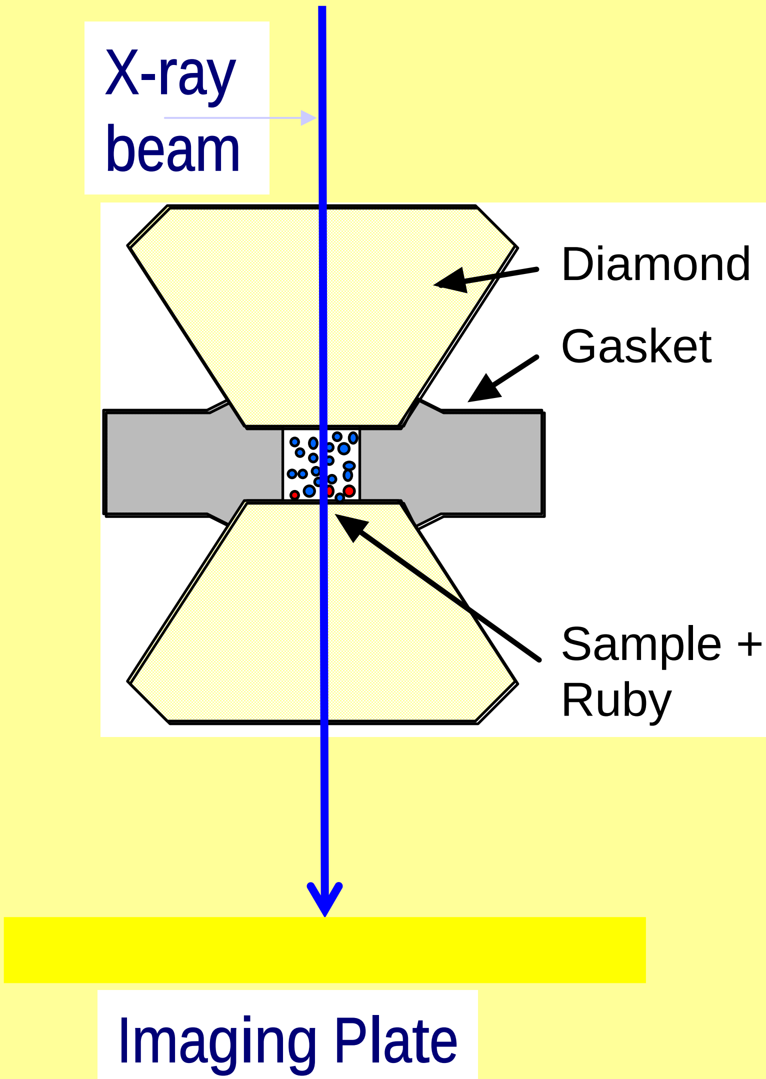
From Jensen



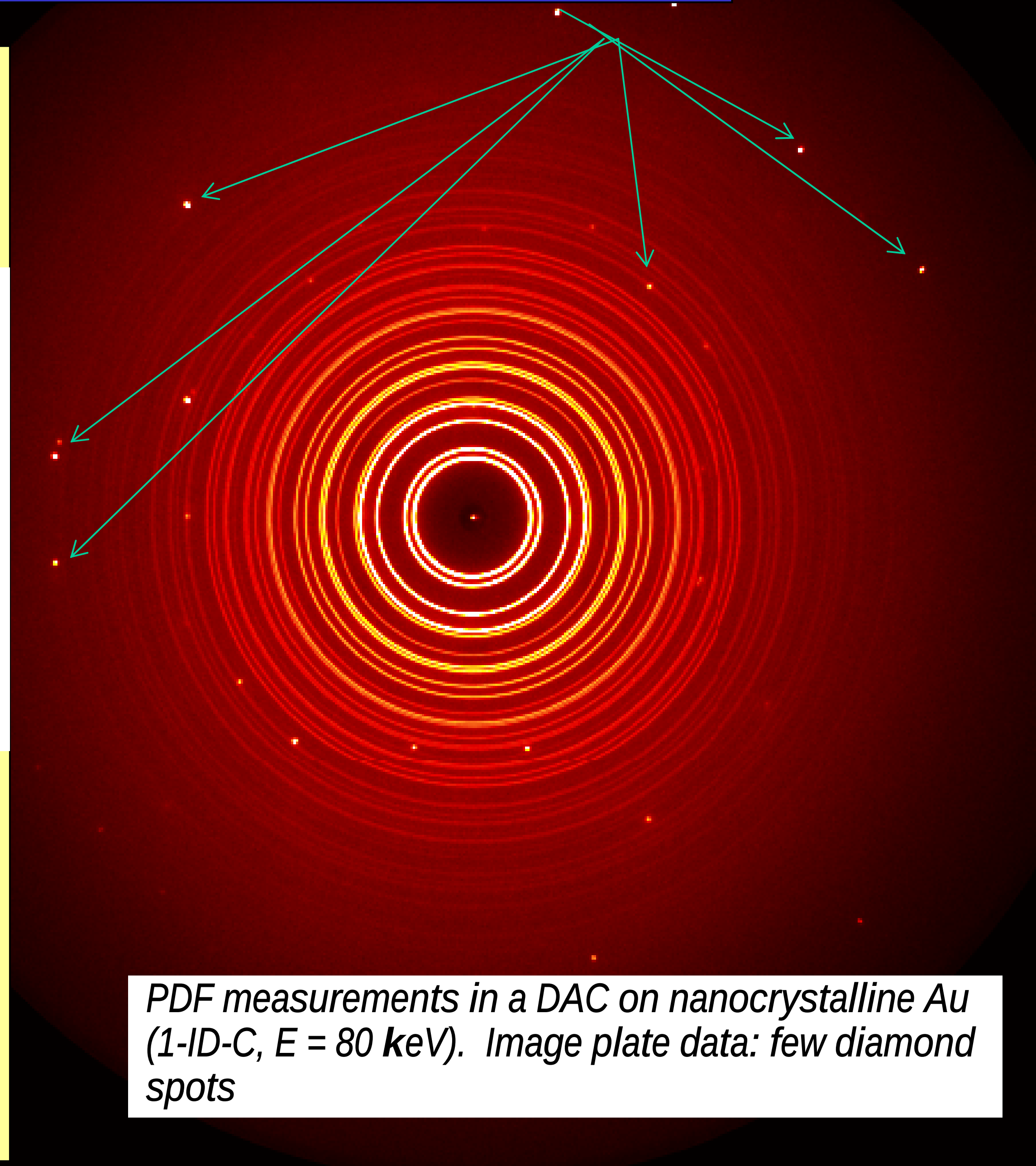
ESRF ID11

Winther, G. Margulies, L., Schmidt, S., Poulsen, H.F., 2004, Lattice rotations of individual bulk grain Part II: Correlation with initial orientation and model comparison. Acta. Mater. In press.

High Pressure PDF Measurements



(From Parise)



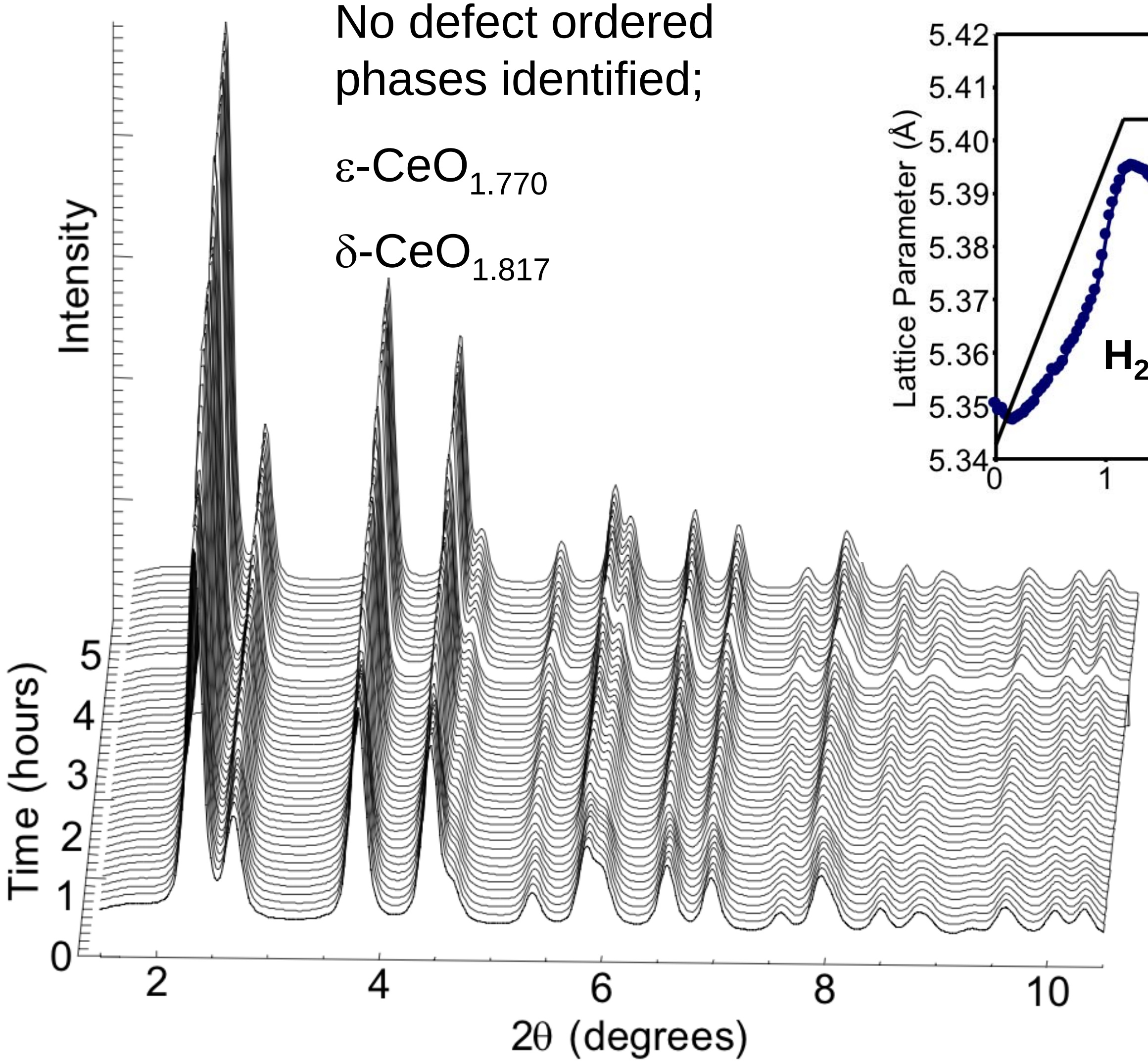
PDF measurements in a DAC on nanocrystalline Au (1-ID-C, E = 80 ***k****eV). Image plate data: few diamond spots*

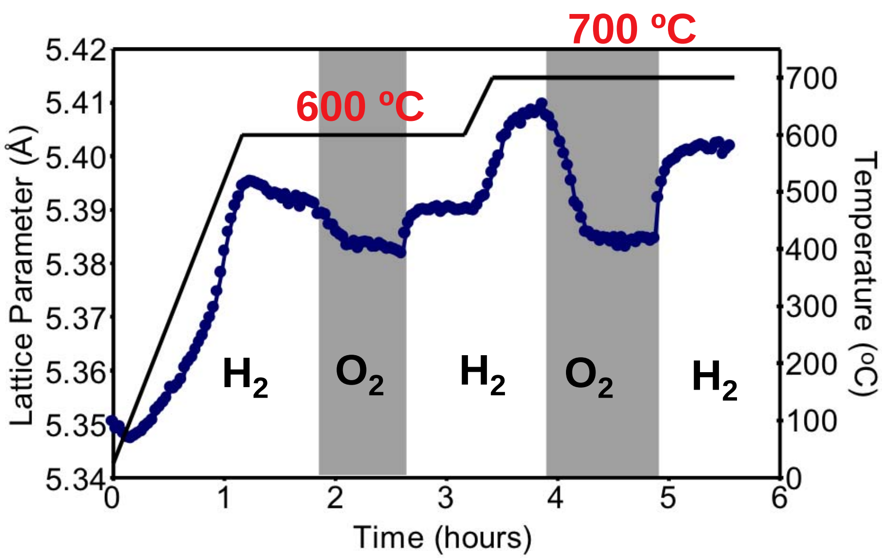
In situ Reduction of Ceria

No defect ordered phases identified;

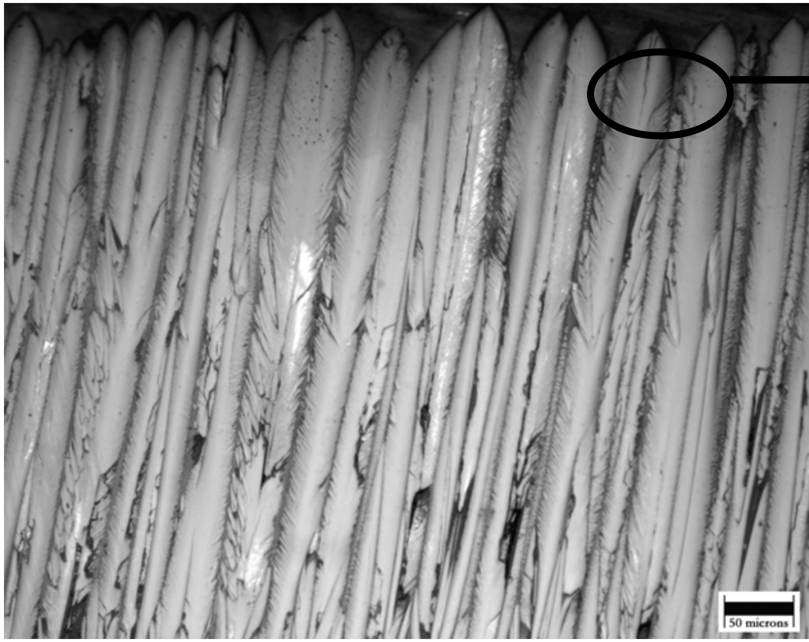
ε-$CeO_{1.770}$

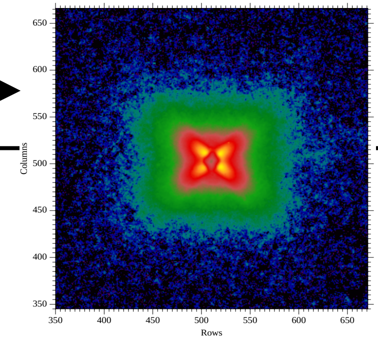
δ-$CeO_{1.817}$



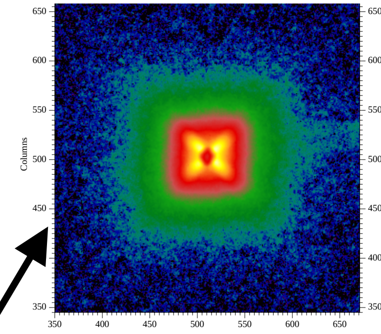


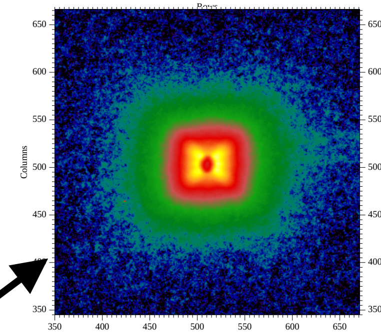
From Chupas

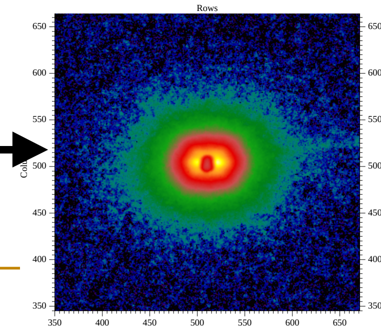


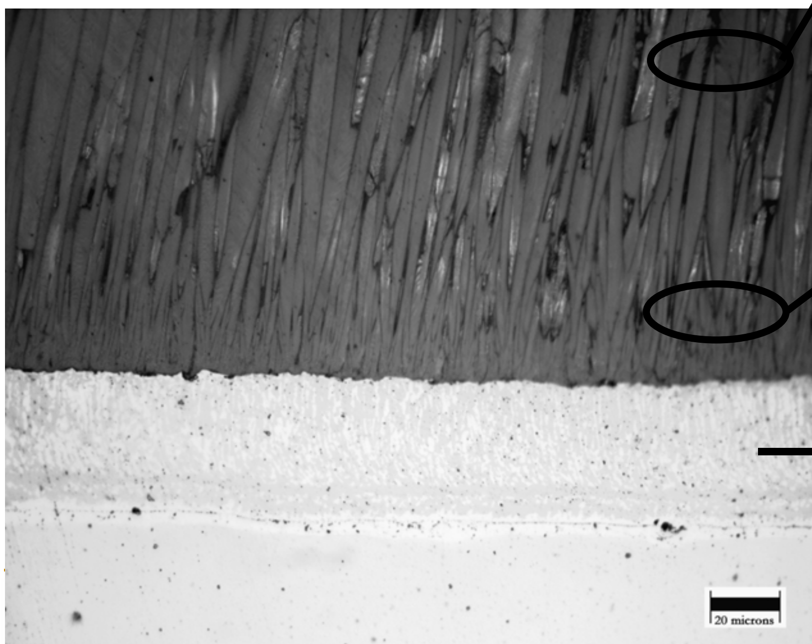


TBC System









•Yttria-stabilized zirconia (YSZ) and bond coat on stainless steel substrate

• Grown using electron-beam plasma vapor deposition, 800μm thick.

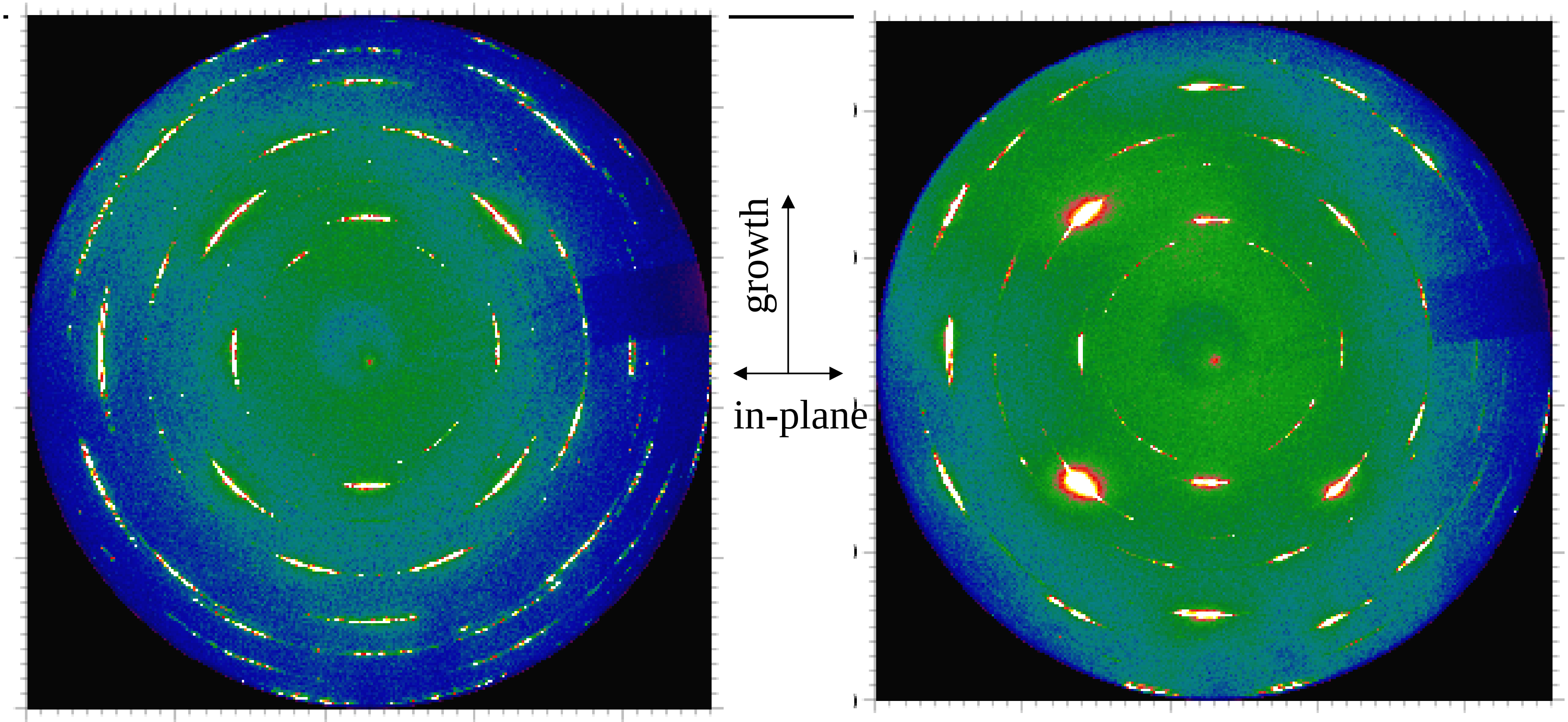
• Porosity affects elastic modulus and thermal conductivity

• Inter-columnar voids and intra-columnar pores (globular and feathery)

• 2-d SAXS with 20μm beam tracks porosity gradient

Almer, Ilavsky, Allen

Wide-Angle Scattering



(2) Interface

(5) Center of Coating

- Tetragonal structure
- Highly textured
- Finer microstructure towards interface

$d_{min} \sim 1$ A

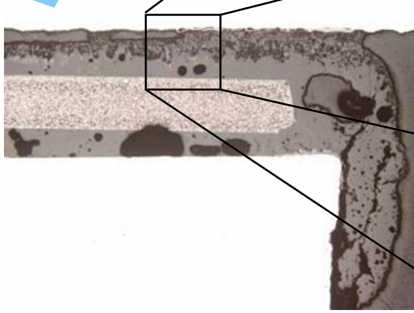
Almer, Ilavsky, Allen

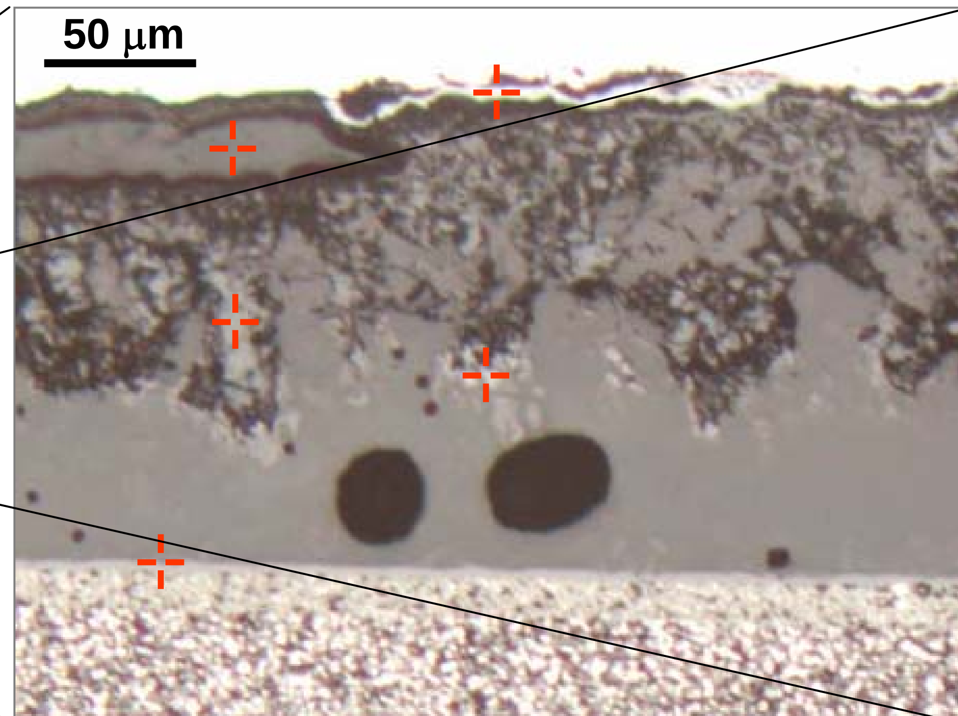
Microdiffraction with high spatial resolution

Cross-section of a SOFC part:

Consisting cathode, anode, electrolytes, interconnect, seal glass,

While elemental information may be obtained by SEM-EDS, it's very important to obtain crystal structure information from region of interest as marked.

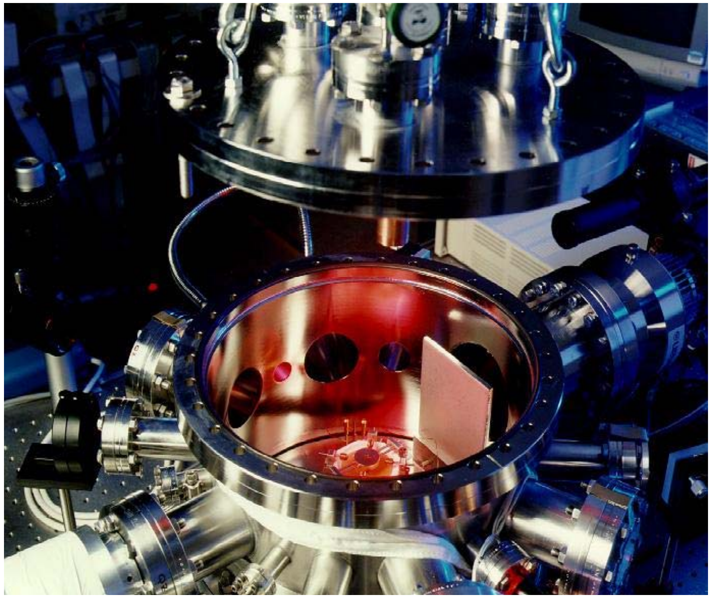




Gao

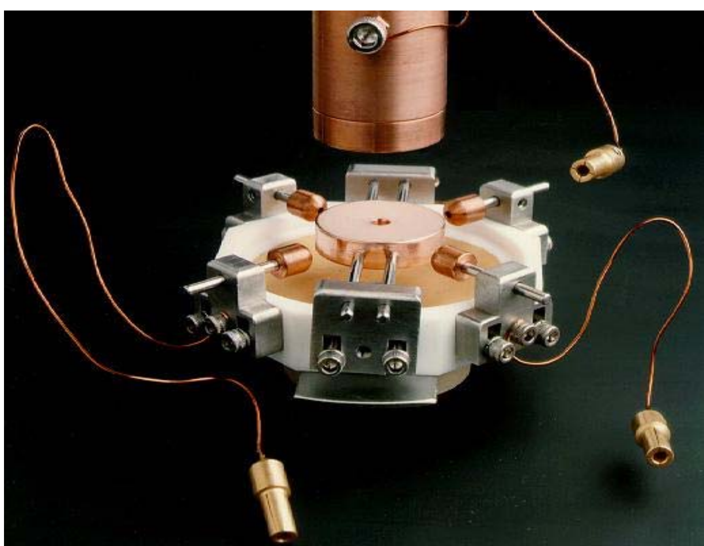
Optical micrograph

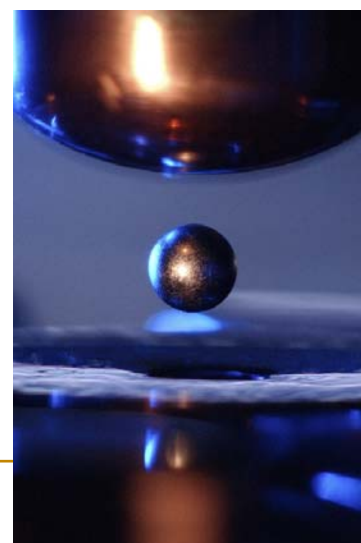
Electrostatic levitation (ESL)
- NASA Marshall Space Flight Center -

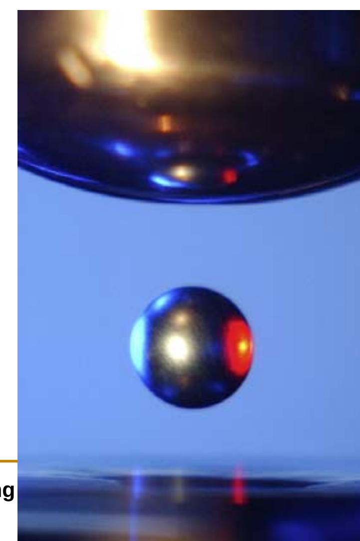


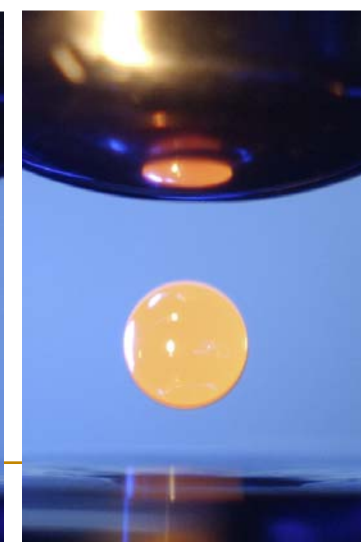
- Sample levitated in high vacuum ($\approx 10^{-8}$ torr)
- Add surface charge on sample by induction
- Maintain surface charge with ultraviolet lamp
- Apply large dc-field to generate sufficient force to counter gravity
- Fast feed-back mechanism to stabilize sample position (three independent sets of electrodes for x, y, and z positioning)
- Lasers used to heat the sample

Goldman

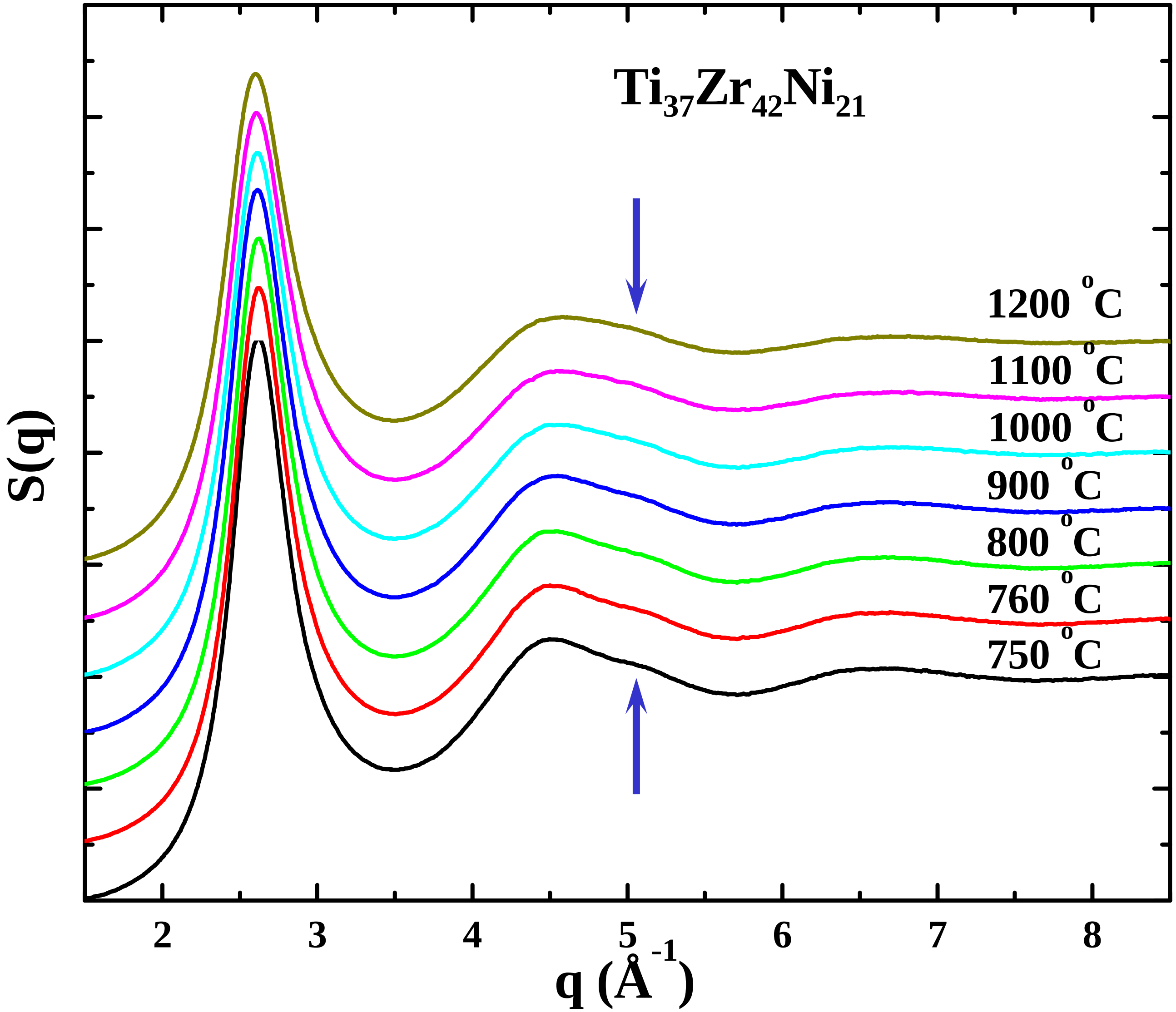








BESL Measurements of S(q) for $Ti_{37}Ni_{42}Ni_{21}$



Increased order with decreasing temperature

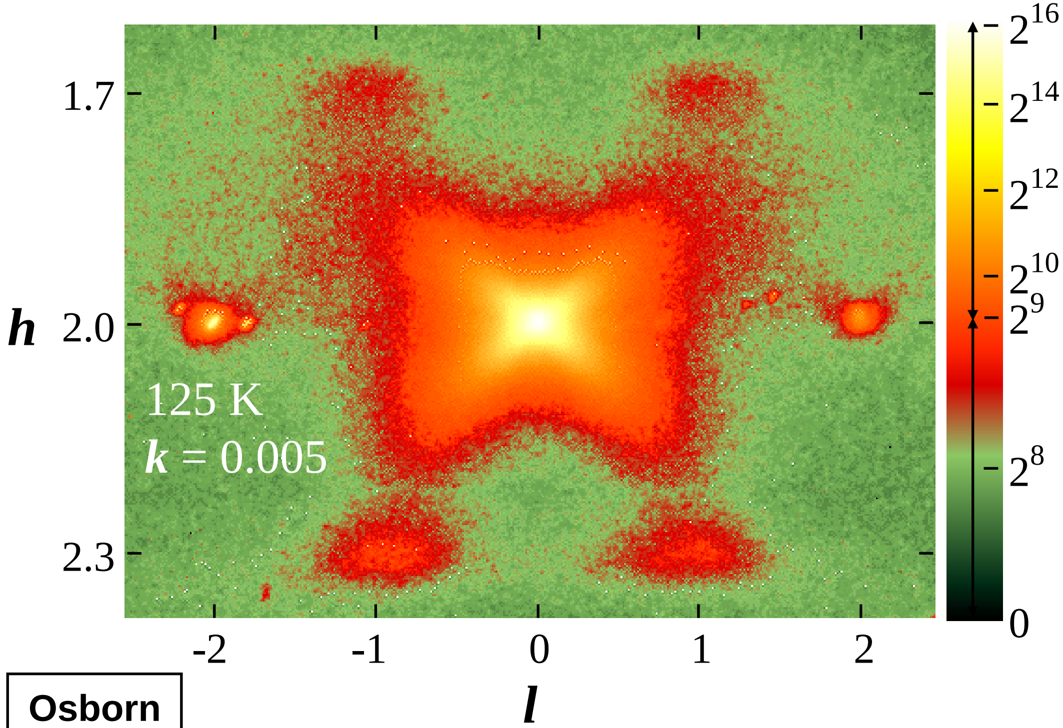
Coordination number is 12 ± 1

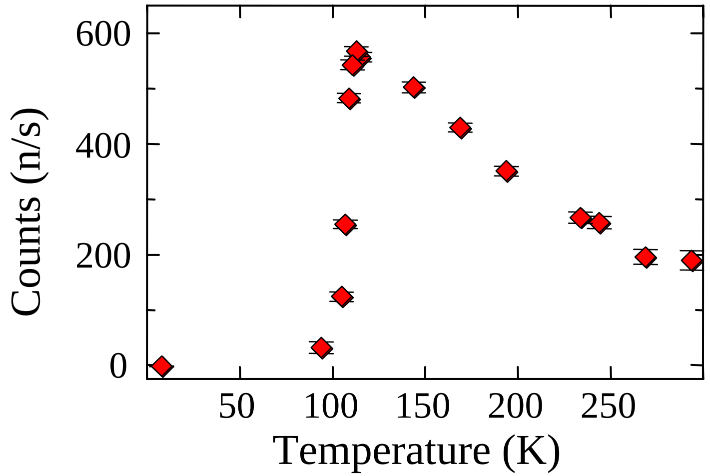
growing icosahedral order

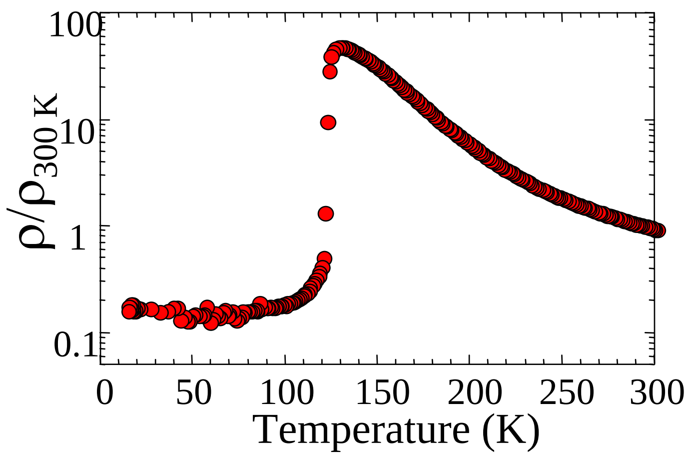
*K. F.Kelton, A. K. Gangopadhyay, G. W. Lee, R. W. Hyers, R. J. Rathz, J. Rogers, M. B. Robinson, D. Robinson, Phys. Rev. Lett, **90**, 195504 (2003).*

Evidence of Polaron Correlations

Bruker CCD x-ray data: 115 keV 11-ID-C



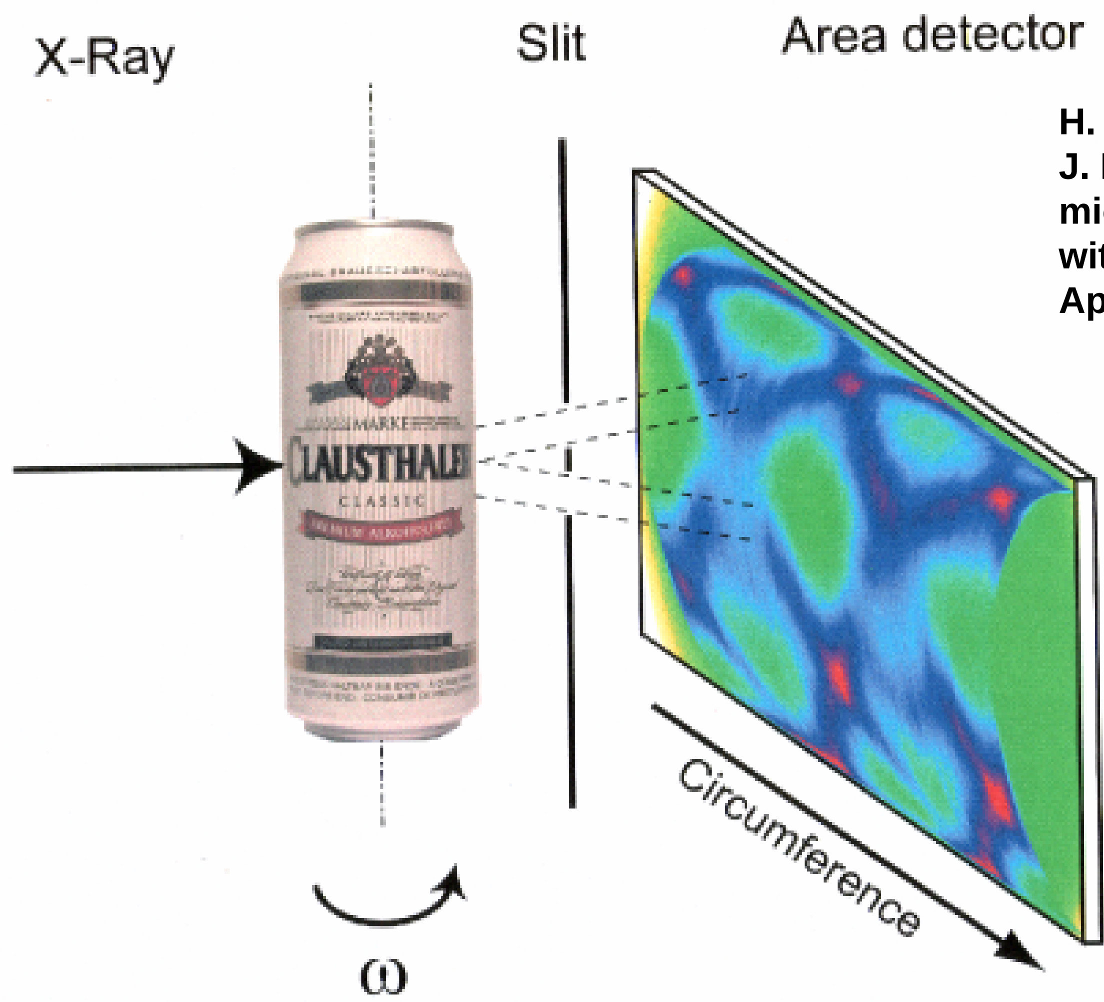




Osborn

L. Vasiliu-Doloc *et al* (PRL 1999)
Shimomura *et al* (PRL 1999)
Adams *et al* (PRL 2000), Dai *et al* (PRL 2000)
Kiriyukin *et al* (PRB 2002)

Texture of Beer Cans



H. J. Bunge, L. Wcislak, H. Klein, U. Garbe, J. R. Schneider (2003). Texture and microstructure imaging in six dimensions with high-energy synchrotron radiation. J. Appl. Cryst.36, 1240-1255.

From Wenk

Charge to the Participants

1. Identify “Grand Challenges” (science and technological) to be addressed during the next 5-10 years which require or high energy x-rays

2. Identify and justify the technical requirements to meet the Grand Challenges

   - New instrumentation and techniques that need be developed on existing beamlines to perform new kind of science.
   - Need for a new dedicated beamline(s) for this community

3. Identify R&D areas that will prepare the community to address the Grand Challenges

Grand Challenges

What are the “Grand Challenges” in science that can be addressed using high-energy x-rays?

Real materials studied in realistic conditions.

Grand Challenges in Mechanics of Materials

- **Need to collect rigorous *in-situ* data at multiple length scales**
- **Integration with mechanics modeling**

Scientific problems to benefit from HE XRD:

- Deformation mechanisms in complex materials (composites, ferroelectrics, etc.)
- *Intra*- and *inter*-granular mechanics
- Microstructure characterization (dislocation structures, etc.)
- Kinetics studies
- Coatings
- Buried interfaces
- Residual stresses (in small structures, components, welds, etc.)
- High-rate deformation? (μsec resolution needed)

Grand Challenges in Structural Science

- **Fast, *in situ* studies of reaction dynamics**
  - Realistic processing conditions
- **Determination of structures in extreme environments**
  - High temperature
  - High pressure
- **Structural information from buried interfaces**

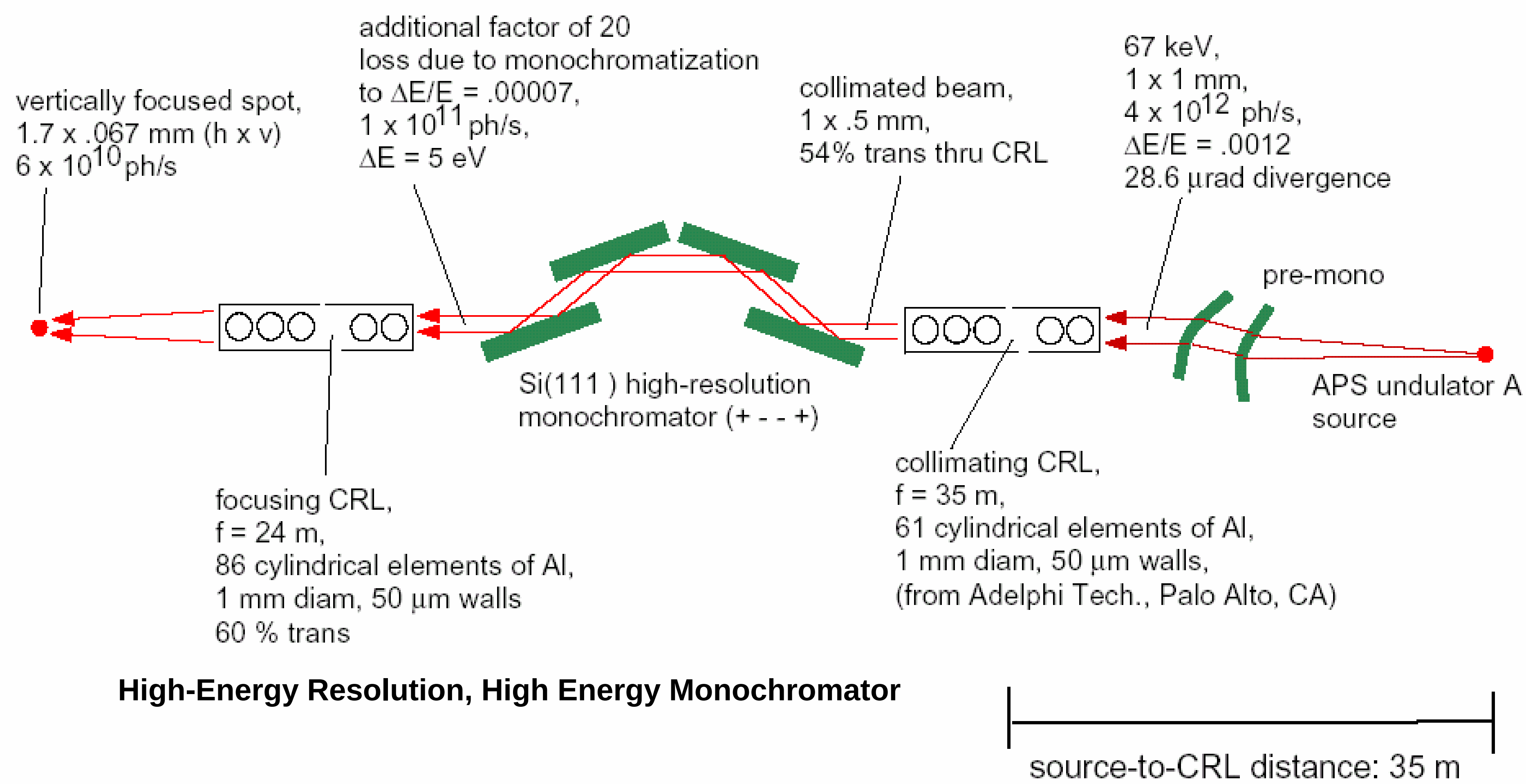
Scientific problems to benefit from HE X-rays:

- Accurate determination of structure for materials containing heavy elements
  - *Low absorption, extinction, polarization corrections*
  - *Use of high-Z K edges for contrast*
- Structures of nanophase materials with rapid PDF techniques
- Studies of bulk materials
  - *Defects using diffuse scattering*
  - *Variation in chemistry/structure bulk vs. surface in concrete*

Technical Challenges

- Dedicated facilities (specialization at APS)
- Faster detectors
- Multiple simultaneous capabilities (imaging, SAXS, texture, etc.)
- Experiment simulation
- Mechanics modeling (and integration with XRD)
- Software for fast and easy data analysis
- Versatile ancillary equipment
- Detailed instrument studies (data integrity)
- Reduced sampling volume
- White beam capabilities
- High energy bend magnet station
- User education

High Energy Resolution Monochromator



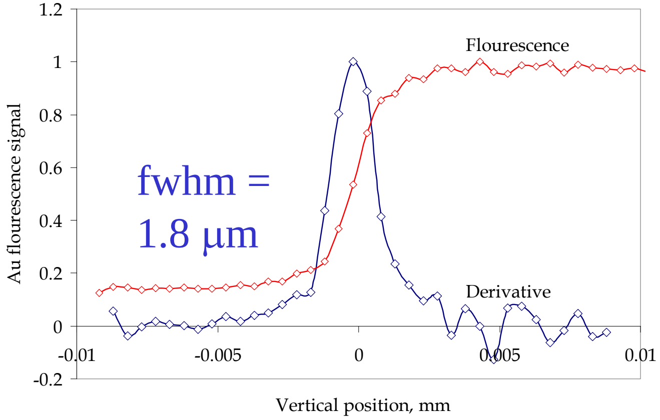
High-Energy Resolution, High Energy Monochromator

S. D. Shastri et al.

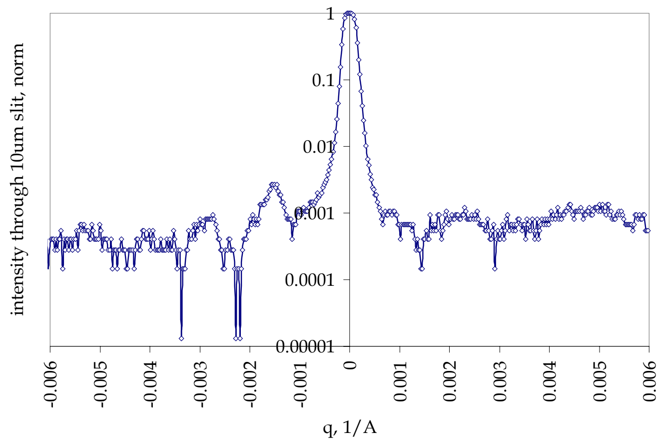
Focusing Optics: Refractive Lenses

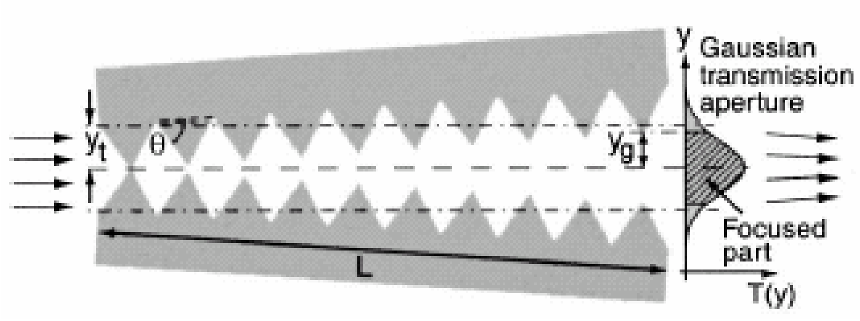
Si sawtooth lens
+ In-line
+ Energy & focal length tunable
- aperture ~ λ^2
- chromatic

Knife-edge profile



Slit profile (wider range)



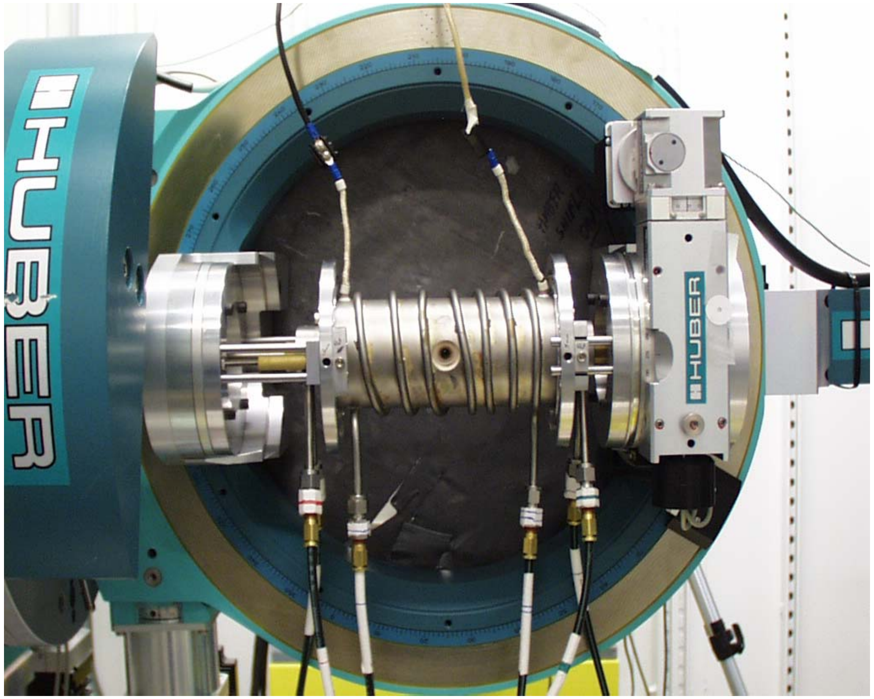


B. Cederström, M. Lundqvist, C. Ribbing, Appl. Phys. Lett. **81** (2002), 1399

High Energy X-ray Undulators

Sample environment

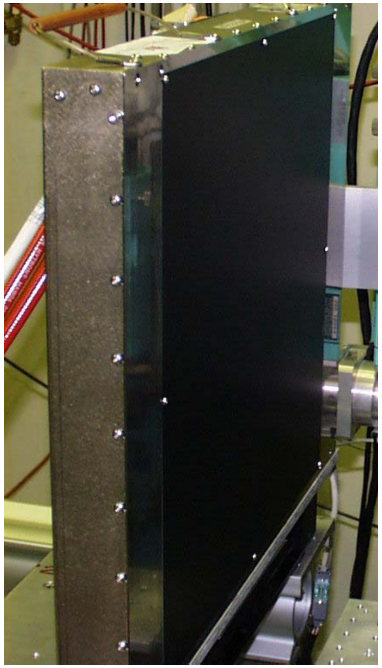
- Fast data acquisition
- Good S:N
- Furnace Design
  - **Eulerian Cradle**
  - **Very low lateral and radial thermal gradient (~ ±2°C over 4 mm distance)**
  - **~1800 K**
  - **Inert to Oxidizing**
  - **Sample rotation for improved powder averaging**
  - **Sample Containment**
  - **Uniform Heating**



Detectors! Detectors! Detectors!

Need fast, large area detectors optimized for high energies

Mar345 on-line image plate



GE Rad detector

Bruker 6500 CCD

Meeting the Challenge

Separate the 1-ID imaging and high-energy x-ray programs.

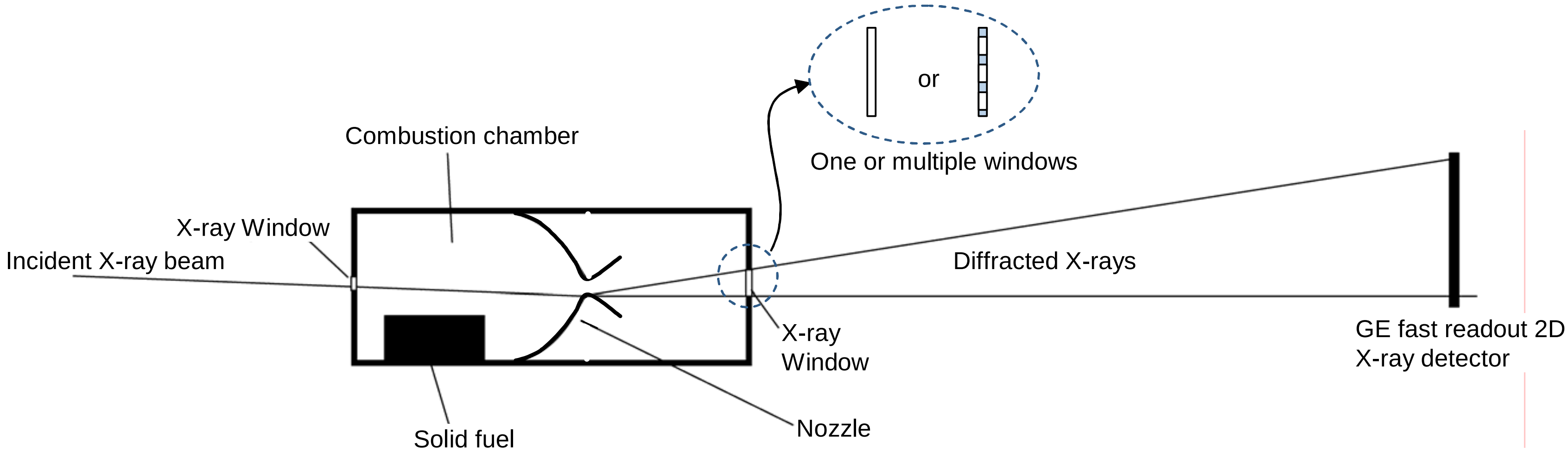
- Move the imaging to a dedicated beamline (~250m)
- Move the high-energy program to a “green-field” sector, or rebuild 1-ID as an optimized high-energy beamline.
- Add a superconducting or optimized permanent magnet undulator
- Curtail ancillary 1-ID activities (need new home for white beam)
- Focus the scientific program on the use of high-energy x-ray brilliance
  - *Microfocusing*
  - *HESAXS*
  - *3D XRDM*

Meeting the Challenge

Optimize 11-ID for High-Energy X-rays

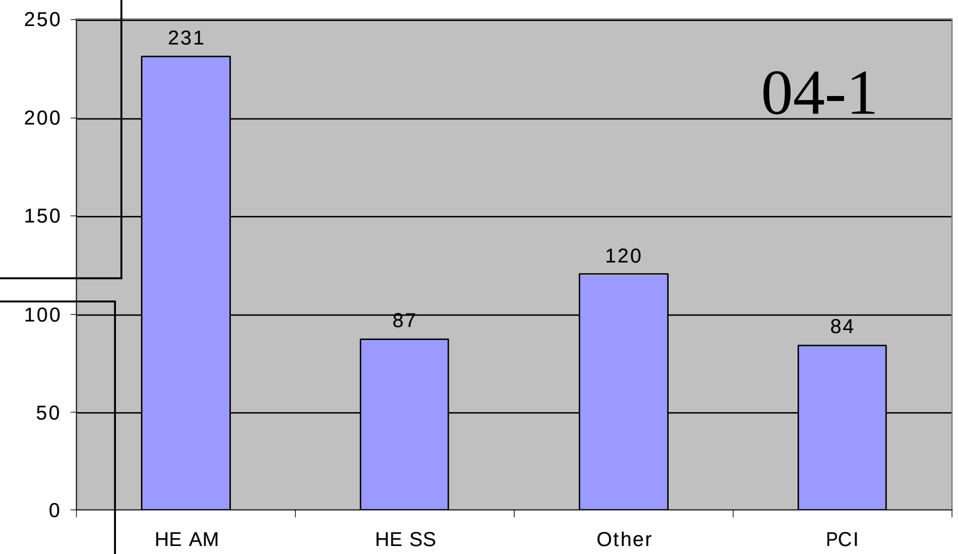
- Replace the EM wiggler with optimized undulator
- Consider canting the beamline
- Upgrade the optics
- Focus program on flux driven experiments
  - *Triple axis diffractometer*
  - *PDF*
  - *Powder diffraction*

Real time study of solid fuel rocket motor nozzle erosion

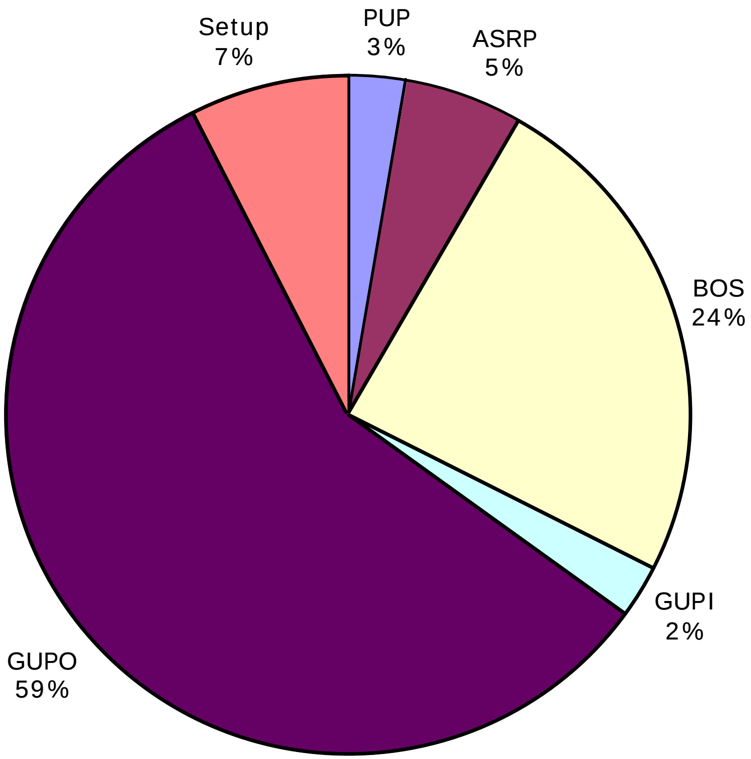


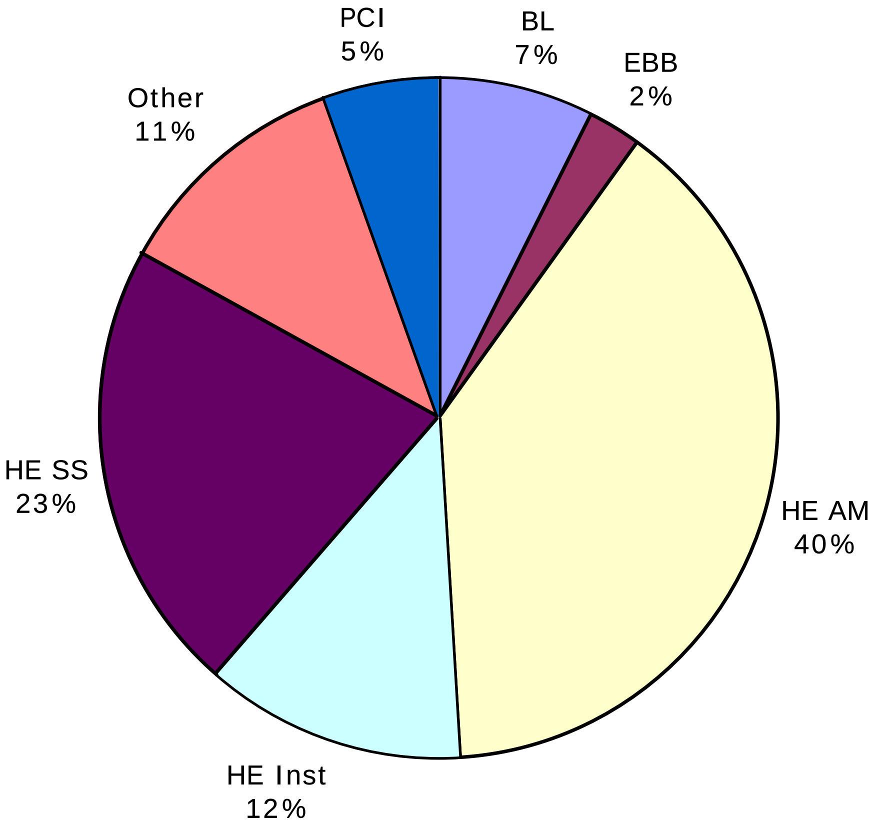
- **Require very high fluxes of high energy x-rays**
  - must penetrate two windows that are subjected to high pressures and temperatures
  - “sample environment” only provides a small angular opening
- **Need very rapid read out detectors**

1-ID Beam Time Allocation

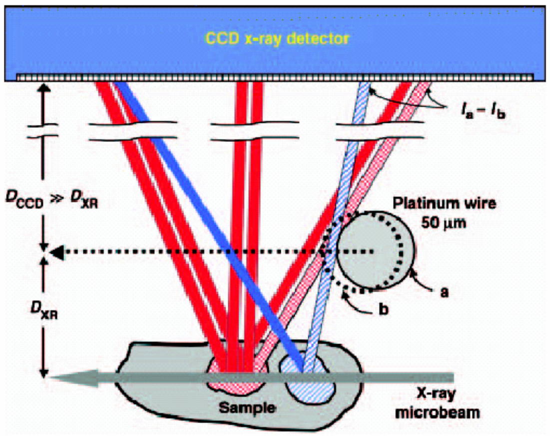


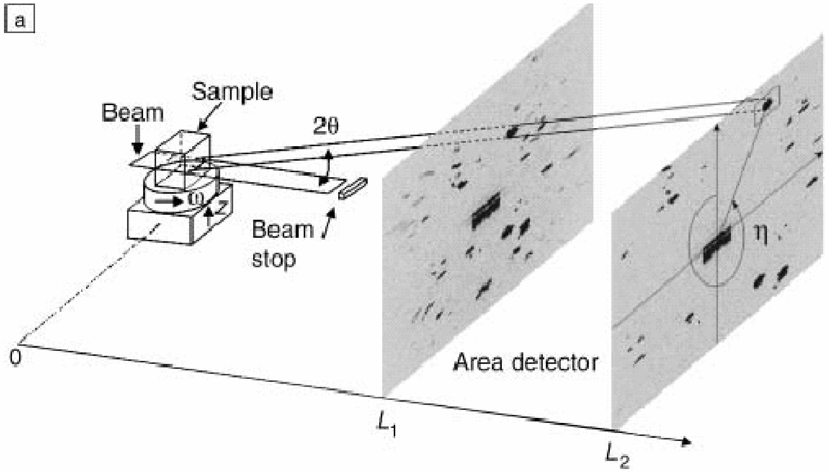
1-ID Beam Time Distribution





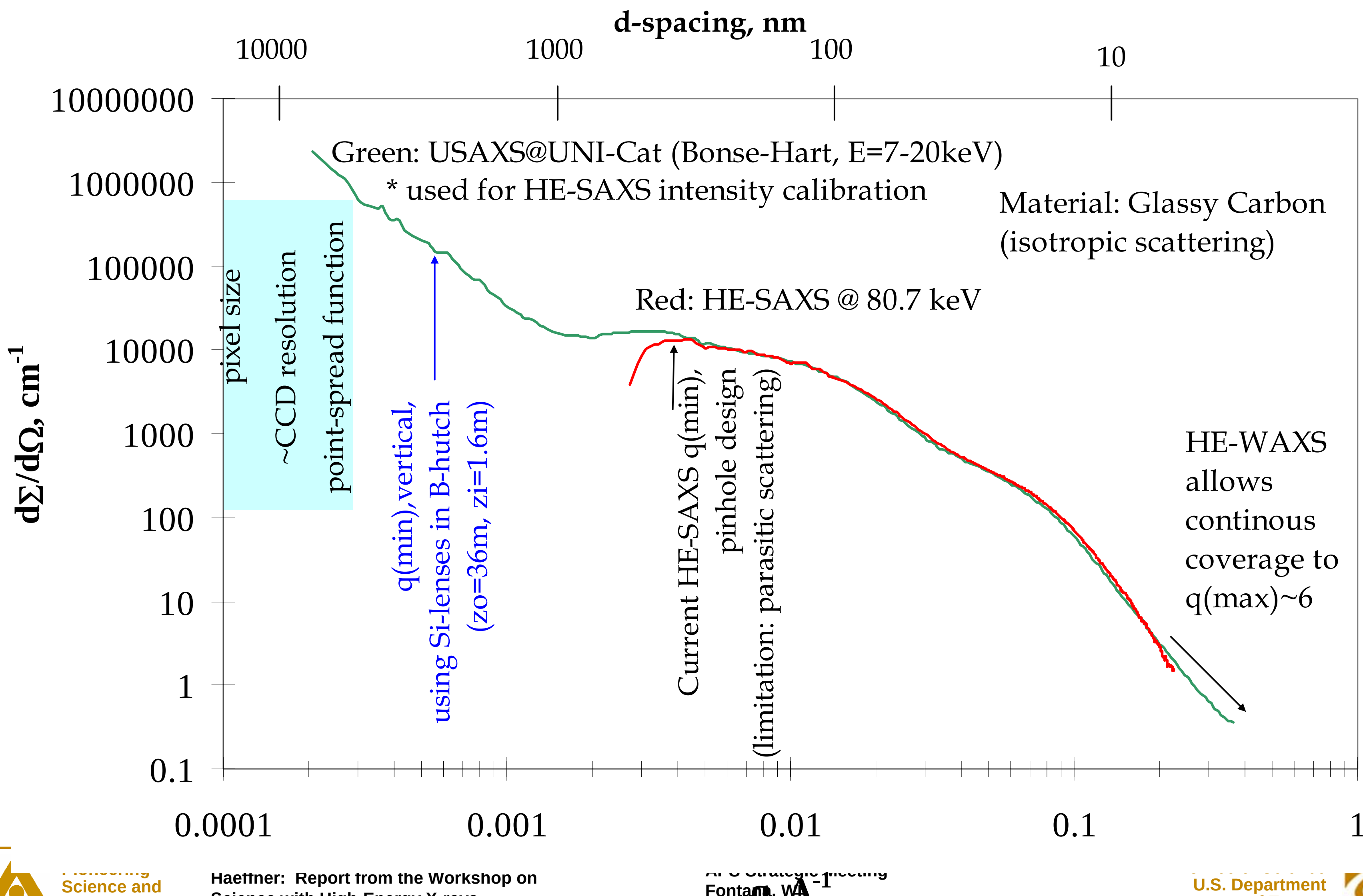
Polychromatic Microbeams*	**High-Energy X-Rays***
G. Ice, B.C. Larson, ORNL	H.F. Poulsen, D.J. Jensen, Risø (DK)
APS UNICAT	G.B.M. Vaughan, ESRF (F)





Energy:	8-20 keV	50-100 keV
1/μ (Al, Fe, Pb):	0.38, 0.018, 0.007 mm	17, 1.9, 0.32 mm
Technique:	polychr. microbeam	monochr. crystal rotation
Resolution:	0.5-1 μm	1-5 μm
3D mapping:	slow	fast

*MRS Bulletin, March 2004



d-spacing, nm
10000
1000
100
10
10000000
1000000
100000
10000
1000
100
10
1
0.1
0.0001
0.001
0.01
0.1
1
q, Å-1
dΣ/dΩ, cm-1
Green: USAXS@UNI-Cat (Bonse-Hart, E=7-20keV)
* used for HE-SAXS intensity calibration
Material: Glassy Carbon
(isotropic scattering)
Red: HE-SAXS @ 80.7 keV
pixel size
~CCD resolution
point-spread function
q(min), vertical,
using Si-lenses in B-hutch
(zo=36m, zi=1.6m)
Current HE-SAXS q(min),
pinhole design
(limitation: parasitic scattering)
HE-WAXS
allows
continous
coverage to
q(max)~6